PRODUCTION OF METAL POWDER BY ATOMIZATION

By

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By
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To

My Wife, Sheila

CERTIFICATE

Certified that this work on "Production of Metal Powders by Atomization" has been carried out under my supervision and that it has not been submitted elsewhere for a degree.

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ACKNOWLEDGEMENT

While theorists can work in splendid isolation the experimentalists work needs many hands and minds for successful completion. This work is no exception. First, I wish to thank Dr. S.P. Mehrotra who guided me throughout the course of this work. Many times he saw the chink of light when I was completely in the dark.

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CONTENTS

LIST OF TABLES

LIST OF FIGURES

ABSTRACT

A unit for production of powders of low melting point metals and alloys by atomization technique was fabricated. The main components of the unit were, a muffle furnace, a container, a nozzle, an atomizer, and a stopper rod. The effect of various operating parameters, viz. temperature of the molten metal, atomizing medium, nozzle diameter, and type of metal on the powder were studied. It was observed that, nitrogen as an atomizing medium produced finer and more regular particles as compared to air atomized particles. The effect of an increase in temperature of the molten metal was to reduce the average size of powder particles. Nozzles of smaller diameter also produced finer particles. Lead powder was coarser and more irregular in shape compared to tin powder, produced under similar conditions. Flow rate, apparent and tap densities of nitrogen atomized particles were higher than the air atomized particles.

CHAPTER 1

INTRODUCTION

The science of powder metallurgy is by no means a new one. According to a report by Mayer[1], the basic principles of powder metallurgy were used on an impressive scale by Indian smiths over 2000 years ago. A famous example is the Delhi-pillar which weighs over six tons and has not corroded during last 1600 years of its existence.

During the last three-four decades, specially after the Second World War, the applications of powder metallurgical techniques as a manufacturing process have increased many folds.[2] One of the main users of metal powders is perhaps the automotive industry. In developed countries 2/3rd of total powder manufactured is used in this industry.[3] An average American car contains 10% of its parts like porous bearings, sliding roof, dynamo and clutches made by powder metallurgy. Refractory metal carbide powders are used in making tools for special purposes, like rock drilling, cutting saws and chisel.[4] Components made of tungsten, molybdenum and tantalum powders are used in electric bulbs, oscillators, mercury arc and X-rays tubes. Metal powders of Be, Al, Mg and Zr in the form of solid fuels are used in rockets and missiles because of better physical and

chemical control, light weight, high energy and minimum residue. A nose cone of a rocket is made of silver infiltrated tungsten.

Metal powders have also found applications in atomic energy. In a nuclear industry 'U' fuel elements and control rods of Be, Zr and Hf are made by powder metallurgy techniques. Dentists use gold and silver powders for teeth filling. Iron powder is used in welding and also in enriched cereals, breads, animal foods, vitamins and pharmaceuticals. These are only a few of the many applications that powder metallurgical techniques find in various fields of day to day life. In fact there is no exaggeration in saying a powder metallurgy part to be 'Ubiquitous'.[5]

There are a large number of methods available to produce metal powders. Some of the important ones are listed and briefly discussed in Table 1.1.[6]

Among the techniques mentioned in Table 1.1 atomisation is perhaps one of the most widely used techniques of powder production for low and medium melting point metals due to low investment cost, high production rate, better quality control and relative ease of prealloying.

The technique of atomisation was first patented by a German scientist in 1882. Basically atomization is a process of disintegration of a stream of molten metal

Table 1.1

Comparison of powder quality produced by various techniques[6]

Sl. No.	Method of production	Particle characteristic		Compressibility	Apparent density	Green strength
		Shape	Size range available			
1.	Atomization	Irregular to smooth rounded and dense particles	Coarse to 325 mesh	Low to high	Generally high	Generally low
2.	Gaseous reduction of oxides	Irregular, spongy	Usually 100 mesh and finer	Medium	Low to medium	High to medium
3.	Gaseous reduction of solutions	Irregular, spongy	Usually 100 mesh and finer	Medium	Low to medium	High to medium
4.	Reduction with carbon	Irregular,	Most mesh from 8 down	Medium	Medium	Medium to high
5.	Electrolytic	Irregular, flaky to dense	All mesh sizes	High	Medium to high	Medium
6.	Carboxyl decomposition	Spherical	Usually in low micron range	Medium	Medium to high	Low
7.	Grinding	Flaky and dense	All mesh sizes	Medium	Medium to high	Low

by high pressurized air, gas or water into fine droplets. Since mechanical strength of liquid metal is much lower than that of a solid metal, the forces required to disintegrate the stream of liquid metal are much smaller compared to the forces required to disintegrate a solid.[7] The principle involved in the technique is schematically shown in Figure 1.1.

Strictly speaking the term atomization is incorrect for even the smallest particle contains hundreds of atoms. It is however, generally accepted term popularly and industrially.

The quality of the powder produced using this technique depends on a number of operational parameters such as temperature of molten metal, viscosity of melt, pressure head of molten metal, nozzle design which includes both diameter and nozzle angle and pressure and type of atomizing medium. The parameters affect the quality in terms of size, size distribution and shape.

The objective of the present study is to fabricate a unit for production of powder of low melting metals and alloys by atomization, and study the effect of some of the operational parameters on powder quality.

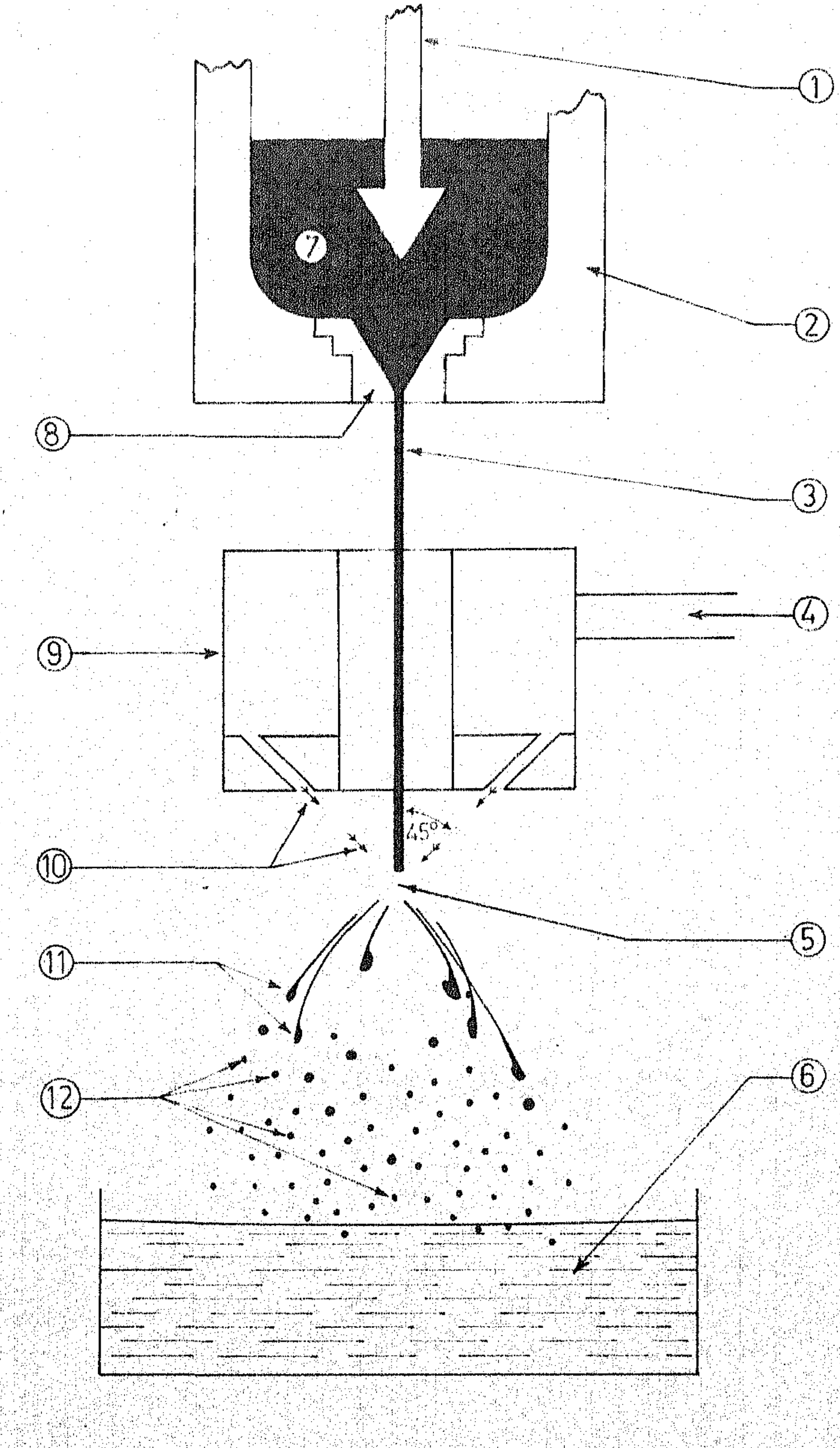

FIG.1.1. PRINCIPLE OF ATOMIZATION (SCHEMATIC)

Figure 1.1: Principle of atomization

(1) Stainless steel stopper rod

(2) Graphite crucible

(3) Stream of molten metal

(4) Air inlet to atomizer

(5) Impingement point

(6) Water bath

(7) Molten metal

(8) Stainless steel nozzle

(9) Stainless steel atomizer

(10) Air jets

(11) Ligaments

(12) Metal droplets

CHAPTER 2

LITERATURE REVIEW

Considerable literature is available on production of metal powders by atomization technique. As has been pointed out in the previous chapter, the quality of powder produced depends on number of operational parameters. Some of the important ones are (1) surface tension and viscosity of molten metal, (2) temperature of molten metal, (3) pressure head of molten metal and nozzle design, (4) atomizer design, (5) atomizing medium, (6) pressure of atomizing medium, and (7) additions.

In this chapter an attempt has been made to present a brief review of literature on the effect of these parameters on the product quality.

2.1 Surface Tension and Viscosity of Molten Metal:

The effect of surface tension and viscosity of molten metal on particle shape and size has been studied by Dixon[2], Thompson[8], Putimtsev[9], and Nichiporenko[10,11]. Thompson[8] found surface tension to be the primary factor governing the size of particle, whereas, Putimtsev[9] and Nichiporenko[10] contended that for molten metal solidifying

at elevated temperature viscosity was the decisive factor. Table 2.1 represents Smithens[12] data on viscosity and surface tension for molten tin and lead at various temperatures. From the data it is evident that at high temperature the viscosity of molten tin and lead approaches that of unity, whereas, an insignificant change in surface-tension was observed. It is therefore more rational to consider surface tension to be more responsible for particle shape and size.

In general surface tension of molten metal varies between five to ten times that of water (73 dynes/cm) and so during atomization fine spherical particles are likely to be formed when surface tension and viscosity are low.

2.2 Temperature of Molten Metal:

Effect of temperature on particle shape and size has been studied by Small and Bruce[13], and Thompson[8]. Small and Bruce[13] found that the likelihood of formation of fine spherical particles increased with increase in melt temperature with water as an atomizing medium. With gas atomization, however, an increase in melt temperature did not exhibit any significant change in particle shape. Similar observation has been reported by Thompson[8] in his study on air atomization of molten aluminium. In general the decrease in surface tension with increase in temperature

Table 2.1

Effect of temperature on surface tension and viscosity of molten tin and lead[12]

Temperature °C	Tin (Sn)		Lead (Pb)	
	Viscosity (n) Poise	Surface tension dynes/cm	Viscosity (n) Poise	Surface tension dynes/cm
300	1.66	560	-	-
400	1.37	555	2.32	463
500	1.18	550	1.85	438
600	1.05	540	1.84	423
800	0.87	-	-	409
1000	-	-	-	-

of molten metal favours the production of irregularly shaped particles but this effect is countered by reduction in viscosity.

2.3 Pressure Head of Molten Metal and Nozzle Design:

Effect of metal head and nozzle diameter have been studied by Dixon[1], and Date and Tendolkar[14]. They reported that an increase in metal head increased the pressure on molten metal passing through the nozzle orifice. With atomizing medium at constant pressure the energy for atomization was constant. Consequently an increase in metal amount yielded coarser particles. Thompson[8] has discussed the effect of nozzle diameter according to him an increase in diameter is likely to increase the particle size directly.

2.4 Atomizer Design:

Huseby[15] has studied the effect of impingement angle of atomizer on particle size. He observed that an increase in angle from 5° to 55° produced finer powders and permitted the use of low water pressure to obtain the same size powder. At an angle greater 55° some backward deflection of atomized particles occurred. Ingram and Turdeller[16] have reported an increase in amount of fine

powder by increasing the angle of water jet from 24° to 42°, the angle of annular gas jet from 13° to 17° and angle of 'V' gas jet from 17° to 28°.

2.5 Atomizing Medium:

A large number of atomizing media viz. air, nitrogen, argon, water, steam, exothermic gases and their mixtures have been used in practice. Jones[7] and Dixon[2] found when molten metal was atomized by air or gas droplets tended to become irregular in shape at the point of impingement but because of high surface tensional forces they had a tendency to become spherical during their fall. In case of water as an atomizing medium, however, the greater distorting forces and severity of quench tended to retain the original irregular shape during their fall. The air, as an atomizing medium tended to form a protective oxide film immediately on atomization which preserved the initial as atomized irregular shape. Lead and zinc for this reason are usually irregular. Connor[17] and Williams[18] produced fine spherical powder using inert and exothermic gases respectively. Werner[19], successfully used a mixture of dry superheated steam and air for producing aluminium powder by atomization.

2.6 Pressure of Atomizing Medium:

Pressure of atomizing medium is another important parameter. The higher the pressure of atomizing medium the higher is the kinetic energy available for atomization at impingement point and this results in higher surface energy of metal powder. Thompson[6] studied the effect of pressure on particle size for air-atomized aluminium powder that an increase in pressure increased the rate of atomization and percentage weight of finer fraction. There is however a threshold value above which an increase in pressure did not increase the fineness of the powder. Suzuki[20] and Ingram[16] have reported an increase in fine fraction of powder with increased gas and water pressure.

2.7 Effect of Additions:

Alloying additions bring about a change in surface tension and viscosity of melt and thereby affect the particle size and shape. Berk[21] discovered that addition of Mg, Ca, Li, Ti and Zr to copper produced irregularly shaped particles when water atomized. Nichiporenko and Naida[22] reported that Mg, Ca, Li, Ti, Mn and Al deformed powder particles by lowering the surface tension of molten copper. Fredorchenko and Nichipornko[23] concluded that alloying additions which lowered surface tension by 30 to 40% did not prevent spherodization.

Nichiporenko and Naida[22] obtained an irregularly shaped air-atomised nickel powder by adding 0.5 wt. % Al. They attributed this irregularity in shape to oxidation of aluminium at the instant of atomisation which raised the viscosity of nickel melt by several orders. This in turn increased the time required for spherodisation resulting in irregularly shaped powder. Tamura and Takeda[24], and Jones[7] suggested that the temperature gap between solidus and liquidus would have some influence on particle shape of atomised alloy powders. Tamura and Takeda[24] have reported an increase in irregularly shaped copper powder on addition of 10% Sn and 30% Pb, which increased temperature gap between liquidus and solidus by 190°C and 600°C respectively. According to Jones, however, a large temperature gap would result in increased time for solidification of metal droplets and therefore longer time for surface tensional forces to form spherical particles.

CHAPTER 3

EXPERIMENTAL

This chapter has been divided into two parts, namely (1) Fabrication of experimental unit, and (2) Experimental technique.

3.1 Fabrication of Experimental Unit:

The essential components of an atomizing unit are:

(1) melting device to melt the metal or alloy to be atomized,

(2) a container to hold the molten metal,

(3) a nozzle through which molten metal is passed,

(4) an atomizer to form an impingement point of high pressurized air jets,

(5) a stopper rod to control the flow of molten metal.

3.1.1 Melting Unit:

In the beginning when the work was started it was decided to use a 12 KVA Induction furnace. After about six months of experimentation the furnace went out of order and so at that stage it was decided to construct a new resistance furnace.

A verticle muffle type resistance furnace with 60 cm O.D. and 37.5 cm height having both the ends open and an actual working zone of 37.5x10x10 cm^3 was constructed. A schematic diagram of the furnace as part of atomizing unit is shown in Figure 3.1. The bottom of the furnace was removable type. The heating element was in the form of a 1/2" diameter coil of 16 gage Kanthal wire. The coil had a total resistance of 30 and was embeded in verticle slots specially made in fire clay bricks. The coil was kept half exposed to the container. The idea of keeping the coil half exposed was to attain as high a temperature as possible without increasing the power input. It could be possible to reach a temperature of about 800°C within a period of four to five hours. Magnesia powder was used to provide insulation packing. Two chromel-alumel thermocouples could be placed at the centre of the furnace, out of which one was connected to a temperature controller and the other to a potentiometer for direct measurement of temperature of molten metal.

3.1.2 Container:

A cylindrical graphite crucible of 7.5 cm diameter and 20 cm length was used as a container to melt and then to hold the metal to be atomized. A two step hole, as shown in Figure 3.8 was drilled. This provided a base on which nozzle rested. The shape of the crucible

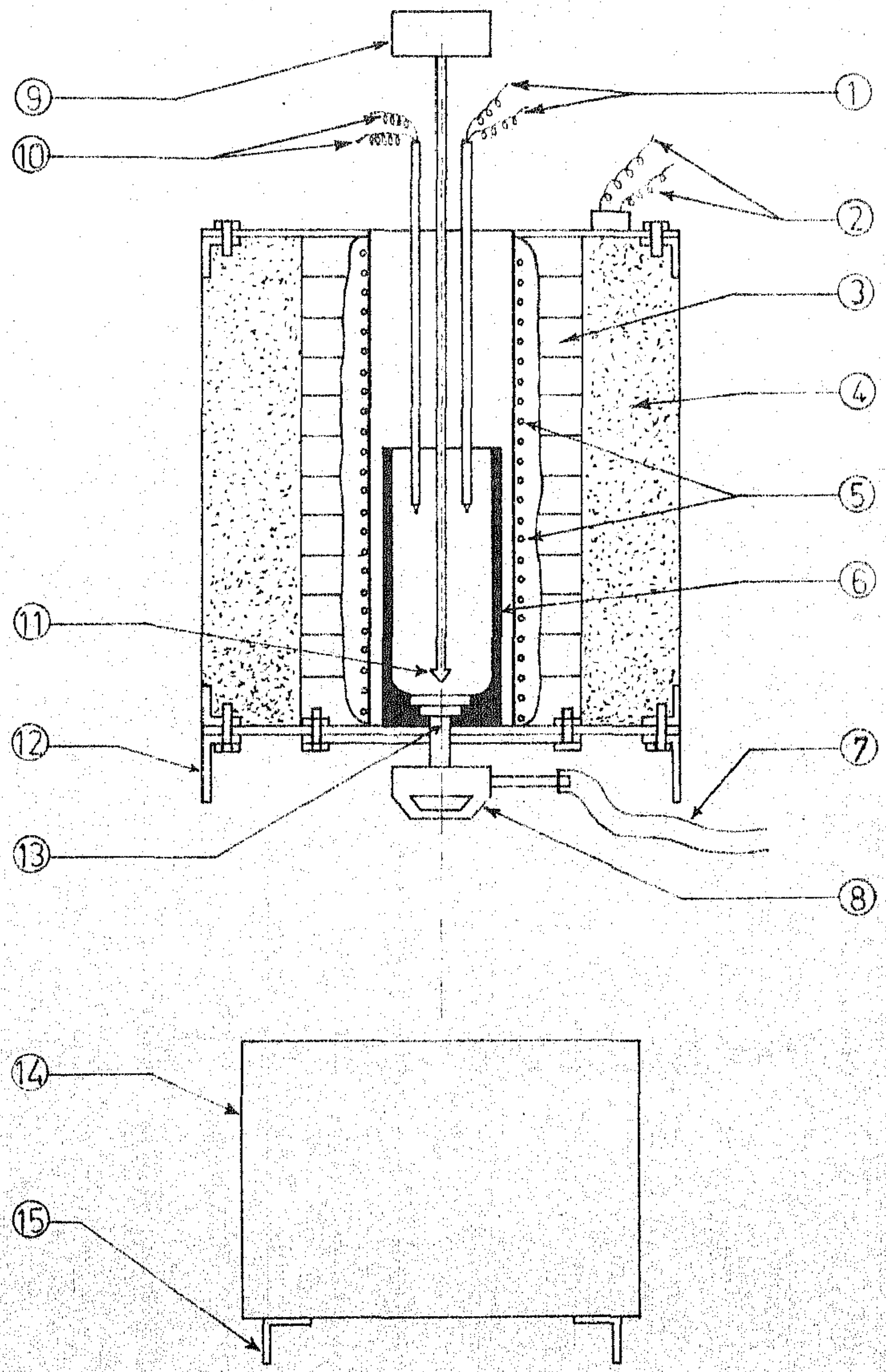

FIG. 3.1. ATOMIZATION SET-UP (SCHEMATIC)

Fig. 3.1. Atomization set-up

(1) & (10) - Chromel-alumel thermocouple
(2) - Furnace input connections
(3) - Fire clay bricks
(4) - Magnesia packing
(5) - Kanthal wire coil
(6) - Graphite crucible
(7) - Rubber tubing for air and gas inlet
(8) - Stainless steel atomizer
(9) - Dead weight
(11) - Stopper rod
(12) - Angles supporting furnace
(13) - Pipe supporting atomizer
(14) - Water bath
(15) - Angles supporting water tank

was found to be an important factor to avoid accumulation of molten metal at the end of the trial. A ceramic coating on the outer crucible lowered the rate of oxidation of the crucible and thus increased its life.

3.1.3 Nozzle and Atomizer Assembly:

Before arriving at the design of the nozzle and the atomizer finally used in the set-up, a number of designs were tried and discarded because of one drawback or the other. Some of these designs and the reasons of their failure are briefly discussed below.

The initial assembly of nozzle and atomizer is shown in Figure 3.2. This arrangement did not work because high pressurized air when passed through the atomizer created tremendous cooling inside the atomizer which was in direct contact with the nozzle. This brought down the nozzle temperature below the melting of metal resulting in premature solidification, and thus hindered the atomization process.

An attempt was made to minimize this cooling effect by providing an insulating ceramic tube inside the nozzle Figure 3.3. Even this did not work because molten metal was still in direct contact with the nozzle at the nozzle tip. The metal got chilled at the nozzle tip and blocked the nozzle opening. A further modification in which both nozzle and atomiser were externally heated

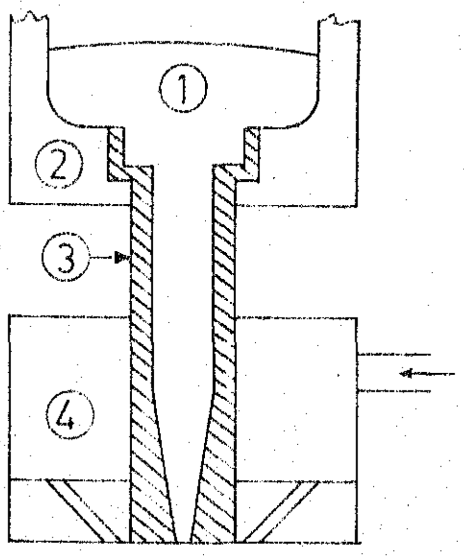

FIG. 3.2

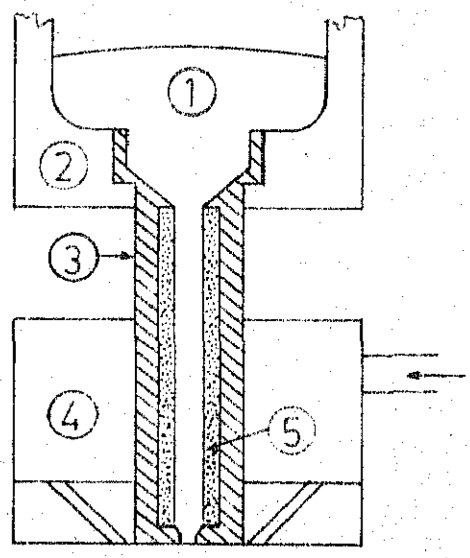

FIG. 3.3

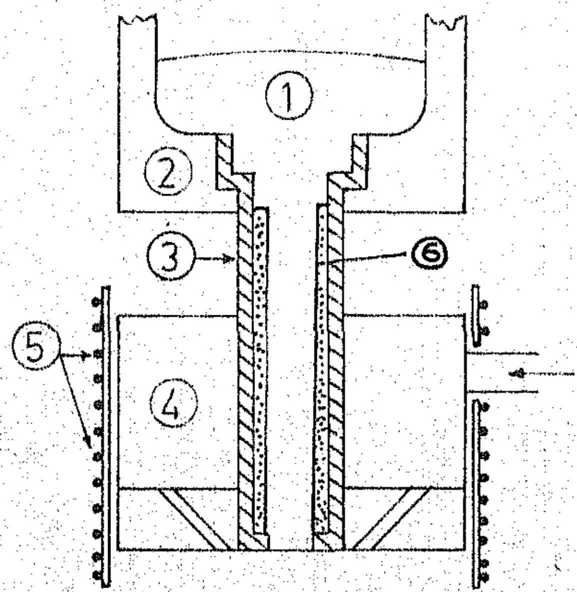

FIG. 3.4

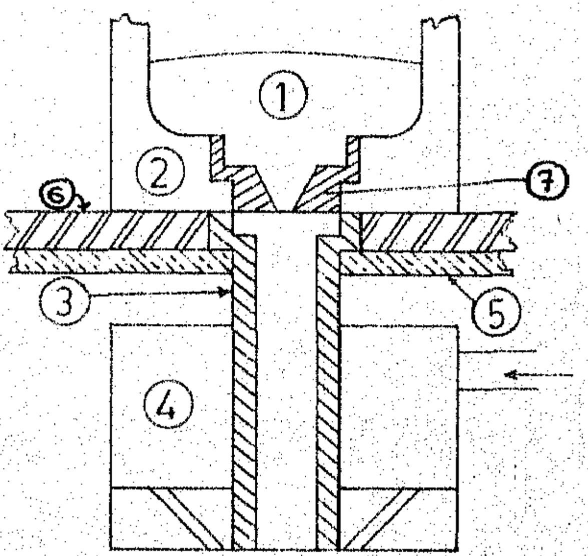

FIG. 3.5

NOZZLE - ATOMIZER ASSEMBLIES

Fig. 3.2. Nozzle atomizer assembly

(1) Molten metal
(2) Graphite crucible
(3) Stainless steel nozzle
(4) Stainless steel atomizer

Fig. 3.3. Nozzle-atomizer assembly with ceramic tube

(1) Molten metal
(2) Graphite crucible
(3) Stainless steel nozzle
(4) Stainless steel atomizer
(5) Ceramic tube

Fig. 3.4. Nozzle-atomizer assembly with external heating arrangement

(1) Molten metal
(2) Graphite crucible
(3) Stainless steel nozzle
(4) Stainless steel atomizer
(5) Resistance furnace
(6) Ceramic tube

Fig. 3.5. Nozzle-atomizer assembly

(1) Molten metal
(2) Graphite crucible
(3) Pipe supporting the atomizer
(4) Stainless steel atomizer
(5) Aluminium sheet
(6) Asbestos sheet
(7) Stainless steel nozzle

using a resistance furnace, Figure 3.4 failed to prevent premature solidification.

At this stage it was decided to separate nozzle completely from atomiser. This design Figure 3.5, although worked at that time, was discarded for it gave an alignment problem. The stream of molten metal did not fall exactly on the impingement point and remained unatomized, unless exactly aligned, which was a problem in itself. All these problems were taken care of to a large extent in a design which was finally arrived at and is discussed below.

3.1.4 Nozzle:

A stainless steel nozzle having a small opening of 1/16" diameter was fabricated as shown in Figure 3.6. The nozzle contained a 'V' shaped cavity which provided a seat for stainless steel stopper rod which when lifted, allowed the molten metal to pass through the nozzle. A small protrusion was made near the bottom of the nozzle for alignment purpose, Figure 3.6.

3.1.5 Stainless Steel Atomizer:

A stainless steel atomizer having six symmetric jets of 1/8" diameter, each inclined at an angle of 45° with verticle axis and meeting at a common impingement point with the verticle stream of molten metal. The

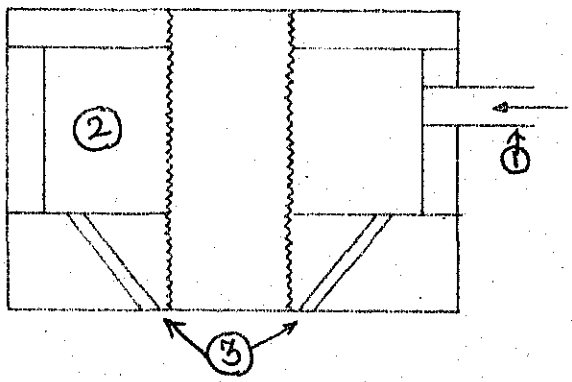

FIG. 3.7. ATOMIZER

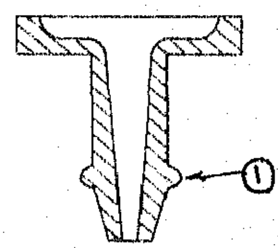

FIG. 3.6. NOZZLE

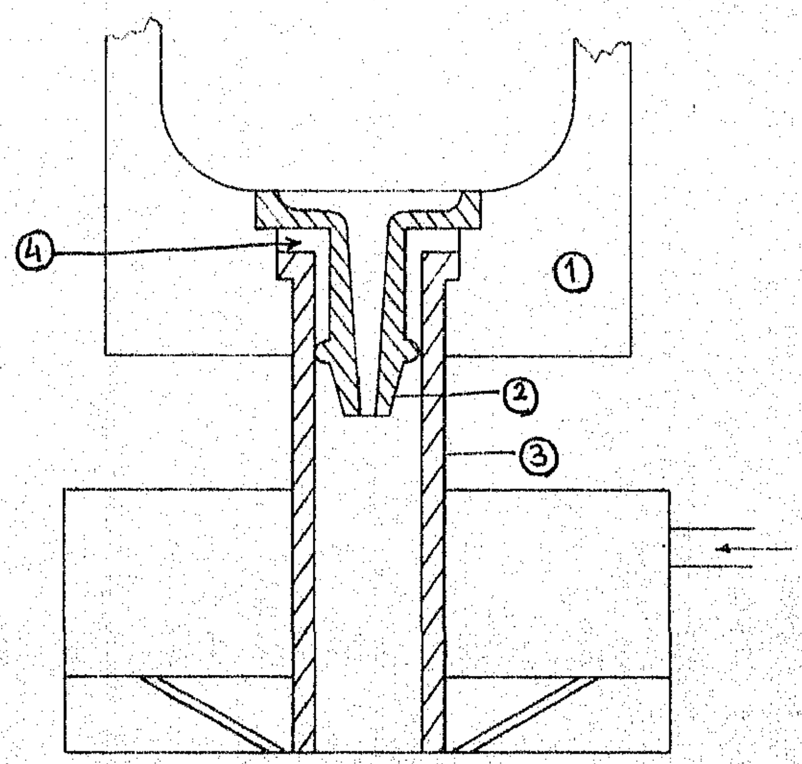

FIG. 3.8. NOZZLE ATOMIZER ASSEMBLY

Fig. 3.6. Stainless steel nozzle

(1) Protrusion for aligament purpose

Fig. 3.7. Stainless steel atomizer

(1) Air inlet
(2) Common space
(3) Air jets

Fig. 3.8. Nozzle atomizer assembly

(1) Graphite crucible
(2) Stainless steel nozzle
(3) Pipe supporting atomizer
(4) Air gap separating nozzle and pipe supporting atomizer

common hollow space in the upper portion of the atomizer maintained uniformity of pressure in each jet. The nozzle atomizer assembly is schematically shown in Figure 3.8.

3.1.6 Stopper Rod:

A stainless steel rod of 8 mm diameter and 400 mm length with a 'V' shaped bottom was used as a stopper rod. The 'V' shaped bottom of the rod fitted in 'V' shaped cavity of the nozzle providing a water tight contact.

3.2 Experimental Technique:

3.2.1 Preparation of Charge:

The charge consisted of small pieces of metal to be atomized and rejected coarse powder and granules from the previous trial. About one kg. charge was used in each run. Before charging the furnace, the charge was properly cleaned, first by hand sorting to remove pieces of metal oxides, ceramic, and other foreign materials, and then by washing with water and acetone to remove dust and clay. The charge was then dried first in air and then in an oven to remove moisture.

3.2.2 Assembly of the Set-up:

Before fixing the crucible the inside of the furnace was properly cleaned and bottom of the furnace was levelled horizontally with the help of a spirit level. The nozzle and the stopper rod were properly cleaned using sand paper to remove oxide layer so as to develop a water-tight contact between the two. The nozzle was placed in the crucible and tested with water to check whether the stream of water passed exactly through the centre of the pipe used for supporting the atomizer. The 'V' shaped bottom of the stopper rod was tightly fitted in the 'V' shaped cavity of the nozzle, and tested with water for any possible leakage. The crucible along with the nozzle and stopper rod was then placed in the furnace which rested on a slotted angle structure at a height of about 3½ feet from the floor. The atomizer was aligned with the nozzle through a pipe which ensured alignment of the metal stream with the impingement point. The atomizer was connected to a compressor or a gas cylinder through rubber tubings. A large water tank was placed exactly below the atomization set-up to collect metal droplets and quench them to room temperature. Schematic diagram of the complete atomizing unit is shown in Figure 3.1.

3.2.3 Procedure:

The prepared charge was fed in the crucible. The two thermocouples used, for controlling, and for direct measurement of temperature of molten metal, respectively, were properly positioned. The top of the furnace was covered with asbestos sheets. A dead weight was fixed on the stopper rod to prevent its up-lift due to metallostatic pressure. The furnace was slowly heated up.

Three sets of experiments were performed. In the first set, tin was atomized using two different atomizing media, namely, air and nitrogen, all other operating conditions being constant. In the second set, the temperature of the molten tin was varied and atomization was carried out using air. In the third set, lead and tin were atomized with air using different nozzle diameters, namely, 5/64" and 1/16". The purpose of these three sets was to study the effect of atomizing media, temperature of molten metal, nozzle diameter and the type of metal on the quality of powder produced.

When the desired temperature was reached and maintained for half an hour, the stopper rod was lifted up and gas valve (for air atomization a compressor was used, and for nitrogen atomisation, a nitrogen cylinder was used) was opened to supply the gas at about 100 psi. The molten metal which came out of nozzle in the form of

thin stream was atomized by gas jets and the fine droplets were collected in the water tank below the atomization unit.

After the run was over the gas switched off and the powder was removed from the tank. The powder was then subjected to various tests after drying.

3.2.3.1 Sieve analysis: Sieve analysis was carried out using a standard set of ASTM sieves. The weights of initial sample and the powder retained on the individual sieves after 15 minutes of sieving were carefully determined. The results of this test are presented and discussed in the Chapter 4.

3.2.3.2 Flow rate of powder: Flow rate is defined as the time required for a known amount of powder (50 gms) to pass through a small opening provided at the bottom of a standard Hall Flow Meter, Figure 3.9. A standard cup of known volume was kept at a distance of 1" from the bottom of Hall Flow Meter, and the time required for the powder to pass through the opening of the hopper was measured accurately using a stop watch.

3.2.3.3 Apparent density test: This test measured the packing density of powder, and was carried out by passing -60+200 mesh powder through a standard Hall Flow Meter so as to fill a standard cup whose volume was accurately

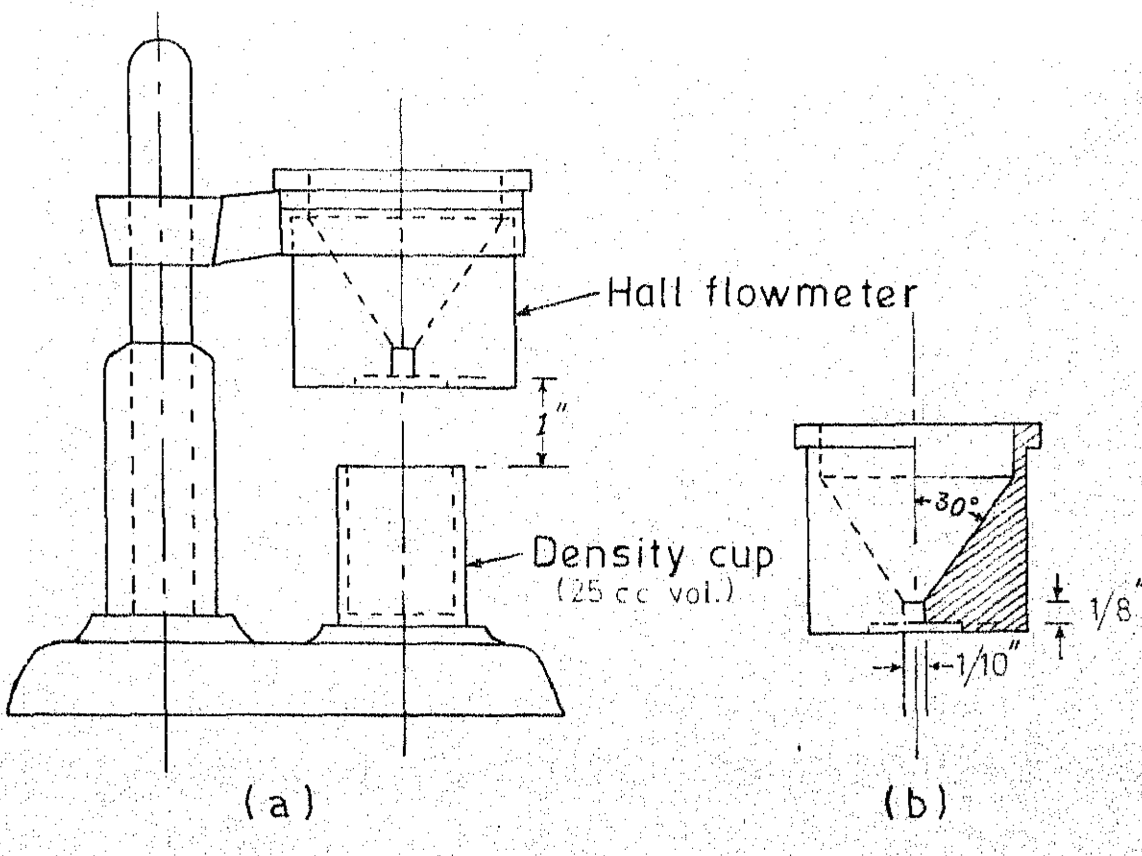

FIG. 3.9. HALL FLOWMETER

(a) With stand (b) Without stand

known. The excess of powder could be removed by levelling it off using a straight edge. Care was exercised, not to disturb apparatus and the cup at all during the test. The powder was then carefully removed from the cup and accurately weighed, knowing the volume and mass of the powder its apparent density could be determined.

3.2.3.4 Tap density: Tap density is the packing density of the powder after it is tapped or mechanically shaken down till the level of the powder in the standard cup falls no more. A -60+200 mesh size sample was taken and fed in the standard Hall Flow Meter and powder was allowed to fall freely in a standard cup of known volume. The cup was constantly tapped until the powder level in the cup became constant. After removing the excess powder the powder in cup was accurately weighed and tap density was determined.

3.2.3.5 Microscopic examination: The effect of various parameters on the shape of the powder particles was studied by observing the powder sample under a microscope at a magnification of 125X. The results are discussed in the following chapter.

CHAPTER 4

RESULTS AND DISCUSSION

As discussed in Chapter 3, the effect of operating parameters on the powder quality was determined by performing following tests:

(1) Sieve analysis,

(2) Flow rate,

(3) Apparent density,

(4) Tap density,

(5) Microscopic examination.

The results of these tests are presented and discussed below.

4.1 Sieve Analysis:

The results of sieve analysis for various sets are tabulated in Tables 4.2 to 4.8.

4.1.1 Effect of Atomizing Medium on Size Distribution:

Size distributions plotted as weight percent of tin retained on individual sieve, for two media, namely, air and nitrogen are shown in Figure 4.1. The shape of the distribution plots in two cases is more or less same. For nitrogen atomized particles, however, there is a trend to shift the distribution curve towards right. This only

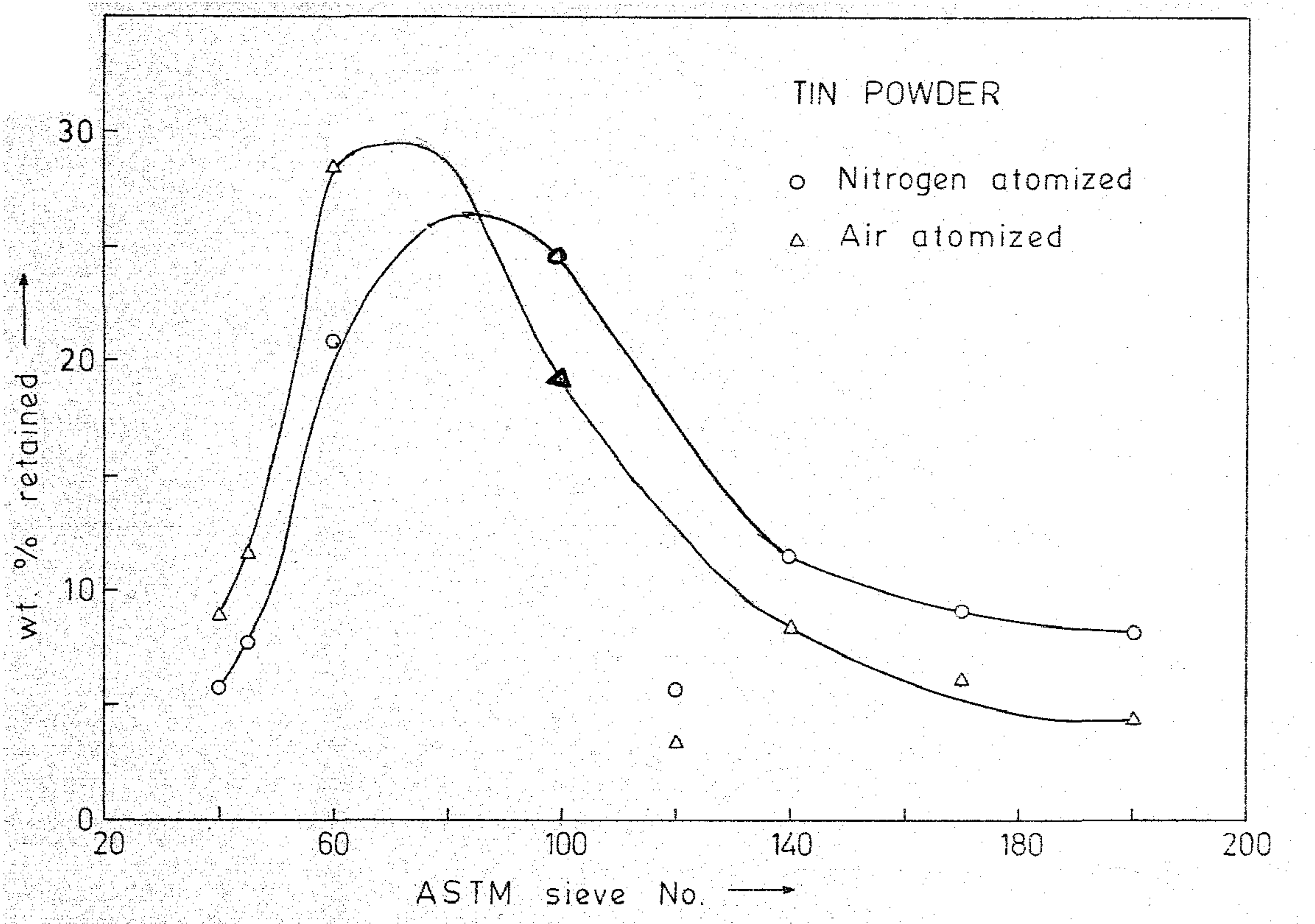

FIG. 4.1. EFFECT OF ATOMIZING MEDIUM ON PARTICLE SIZE DISTRIBUTION.

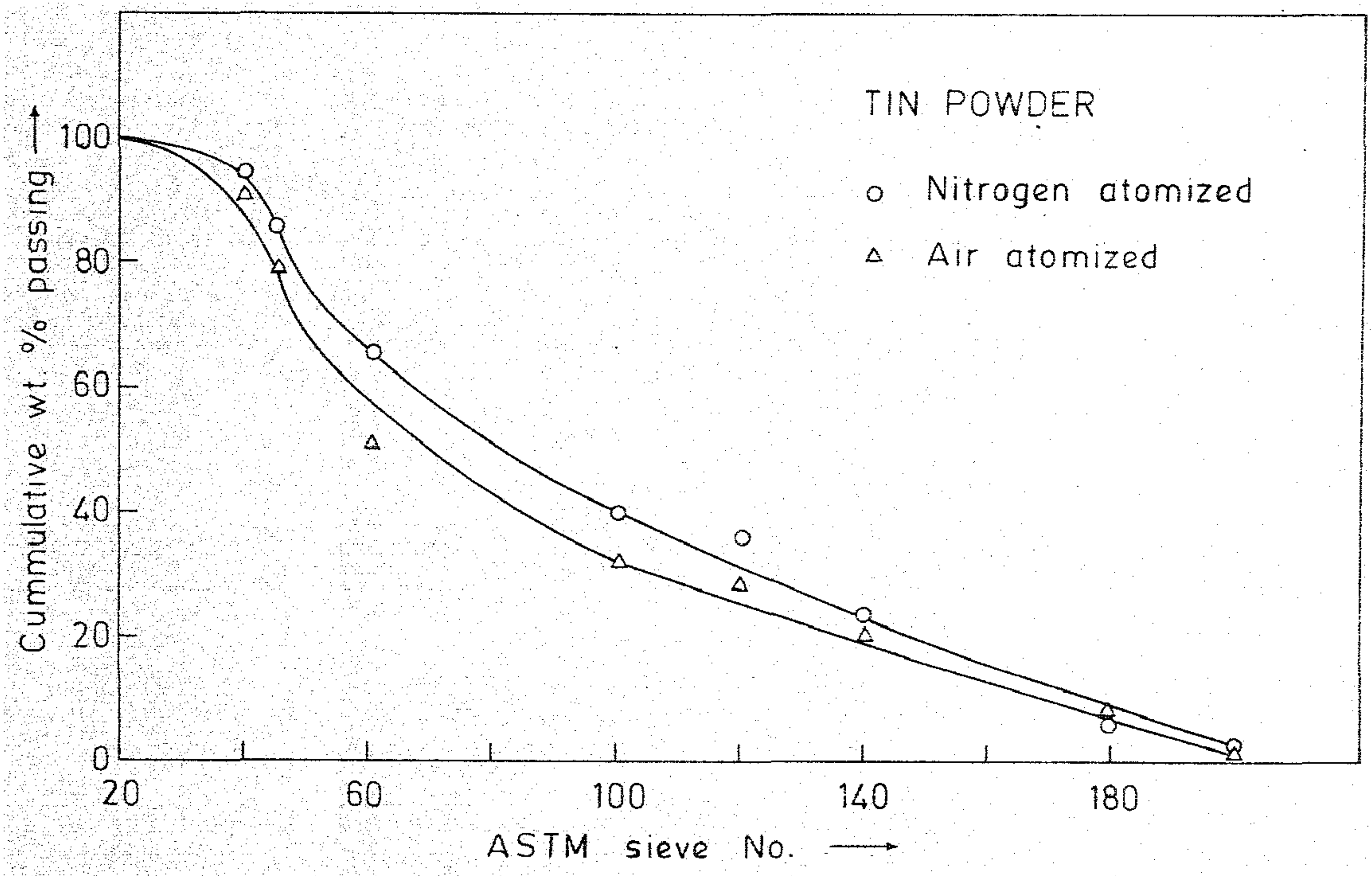

FIG. 4. 2. EFFECTED ATOMIZING MEDIUM ON WEIGHT PERCENTAGE.

Table 4.2

Effect of nitrogen as an atomising medium on particle size distribution of tin powder

Metal : Tin   Medium : Air

Temperature : 650°C   Nozzle diameter : 1/8"

Pressure : 100 psi

ASTM Sieve No.	Opening size in microns	Average opening size in mm	Weigh % retained	Cummulative wt. % passing
+ 40	+420	0.42	5.730	94.270
- 40+45	-420+350	0.385	7.640	86.630
- 45+60	-350+250	0.300	20.830	65.800
- 60+100	-250+150	0.199	24.890	40.910
-100+120	-150+125	0.174	5.650	35.260
-120+140	-125+105	0.114	11.615	23.645
-140+170	-105+88	0.096	9.360	14.285
-170+200	- 88+77	0.081	8.085	6.200
-200+230	- 77+62	0.068	5.125	1.075
-230	- 62	0.062	1.075	-

Average size of powder : 150 micron

Shape : Irregular

Table 4.3

Effect of air as an atomizing medium on particle size distribution of tin powder

Metal : Tin   Medium : Air

Temperature : 650°C   Nozzle diameter : 1/8"

Pressure : 100 psi

ASTM Sieve No.	Opening size in microns	Average opening size in mm	Weight % retained	Cummulative wt. % passing
+ 40	+420	0.42	8.940	91.060
- 40+45	-420+350	0.385	11.620	79.440
- 45+60	-350+250	0.300	28.145	51.295
- 60+100	-250+150	0.199	19.180	32.115
-100+120	-150+125	0.174	3.200	28.915
-120+140	-125+105	0.114	8.315	20.600
-140+170	-105+88	0.096	6.155	14.445
-170+200	- 88+77	0.081	4.360	10.085
-200+230	- 77+62	0.068	6.835	3.250
-230	- 62	0.062	3.250	-

Average size of powder : 166 microns

Shape : Irregular

indicates that the fraction of finer powder is more compared to that in the case of air atomized particles. The average particle size in the case of nitrogen atomized powder is 152 microns, whereas in the case of air atomized it is 166 microns. The production of coarser particles in the case of air atomization can be attributed to formation an oxide coating around the metal droplets. Dixon[2] suggested that oxidation of metal during air atomization increased viscosity of molten metal during air atomization which was responsible for the formation of coarser particles. Figure 4.2 shows a plot of cummulative weight percent passing of air and nitrogen atomized powder. Similar conclusion can be drawn from this plot that, the cummulative weight percent passing, in the case of nitrogen atomized powder is higher than in the case of air atomized powder.

4.1.2 Effect of Temperature on Size Distribution:

The size distribution plotted as the weight percent retained on individual sieve for air atomized tin powder at three different temperatures of molten metal, namely, 650°C, 540°C, and 450°C are shown in Figure 4.3 it is evident that with increasing temperature the size distribution curve shifts to right, the peak of the curve is lowered, and so weight percent of coarse fraction

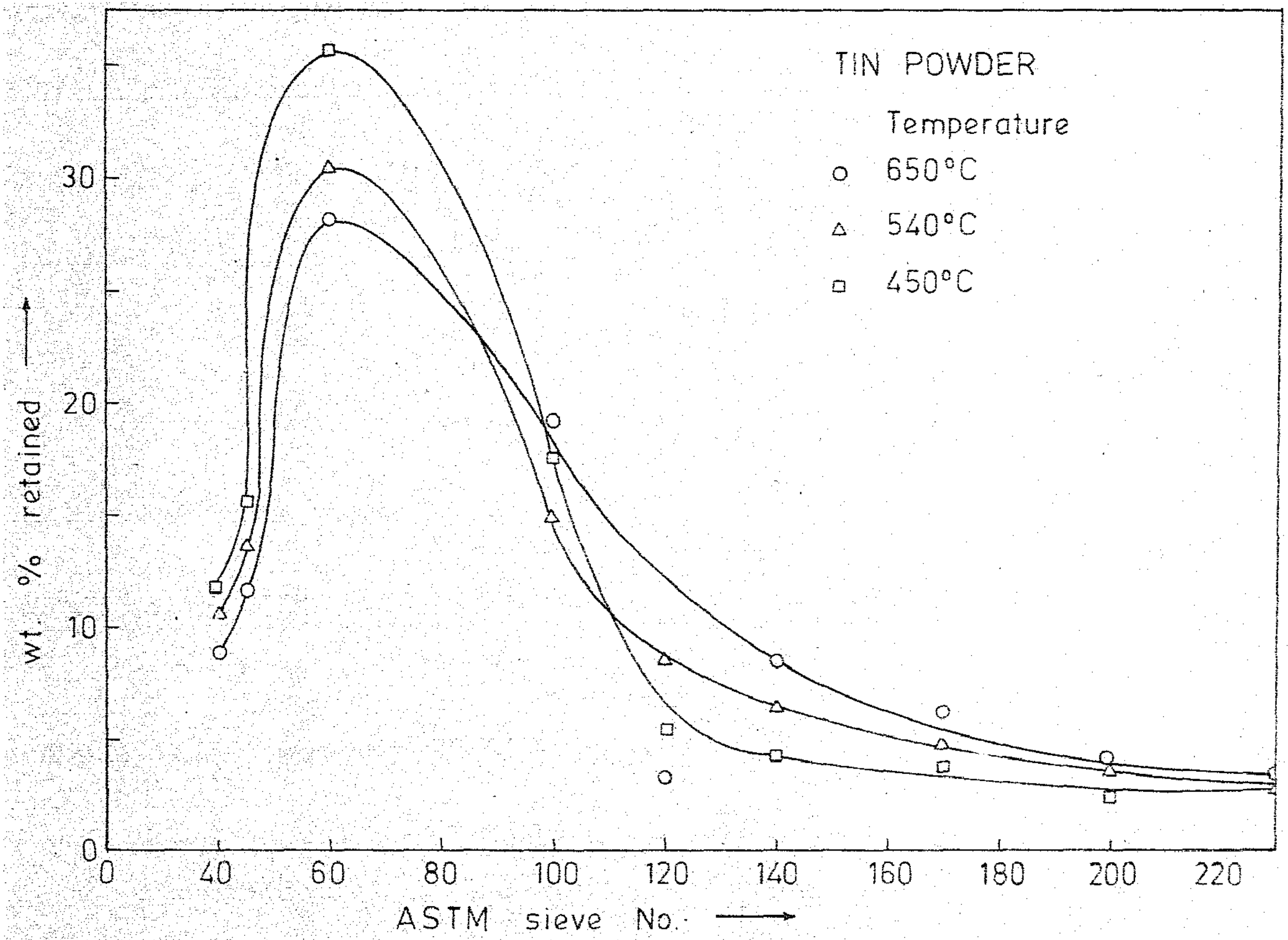

FIG. 4.3. EFFECT OF TEMPERATURE ON PARTICLE SIZE DISTRIBUTION

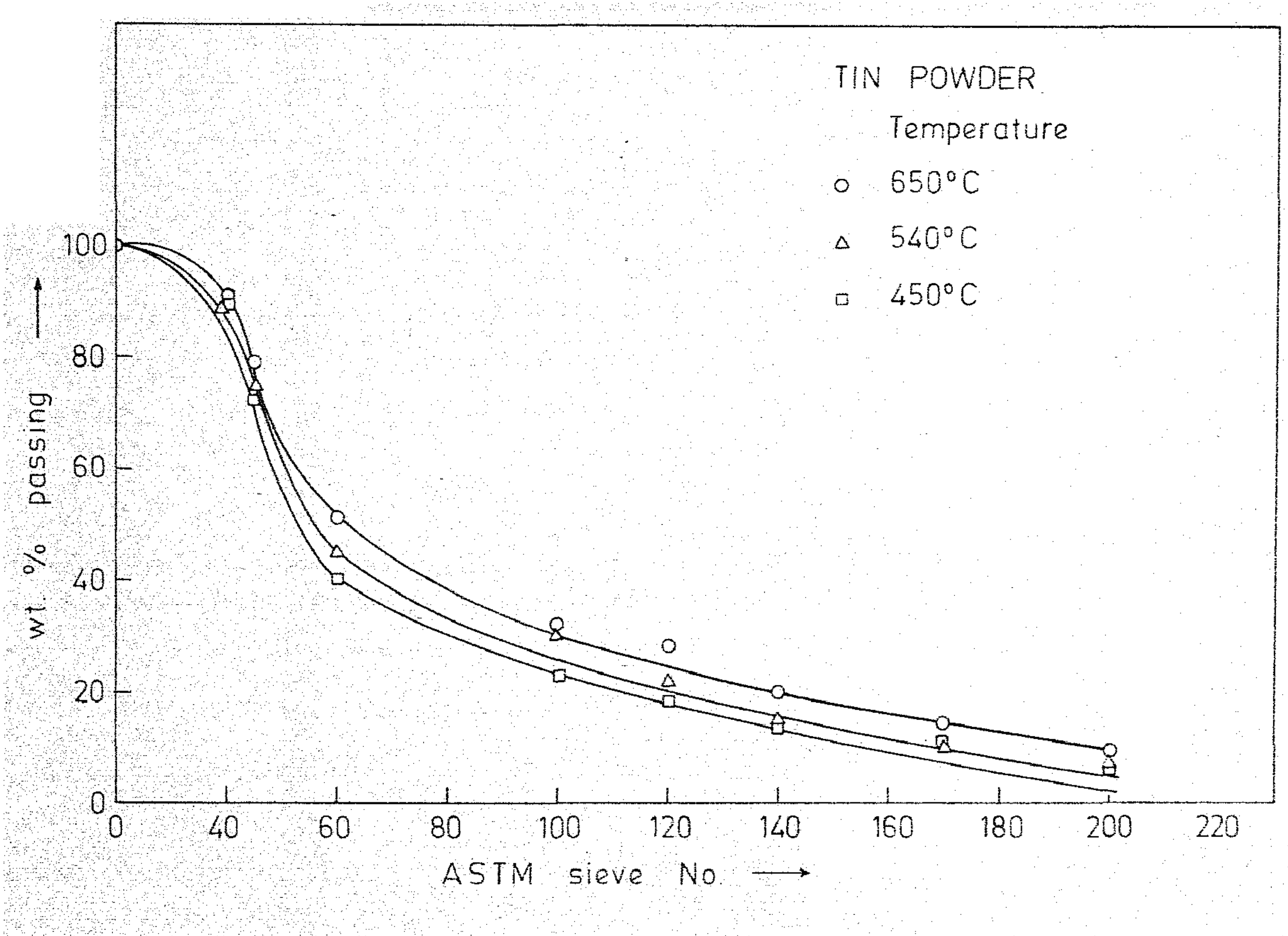

FIG. 4.4. EFFECT OF TEMPERATURE ON COMMULATIVE WEIGHT PERCENTAGE PASSING

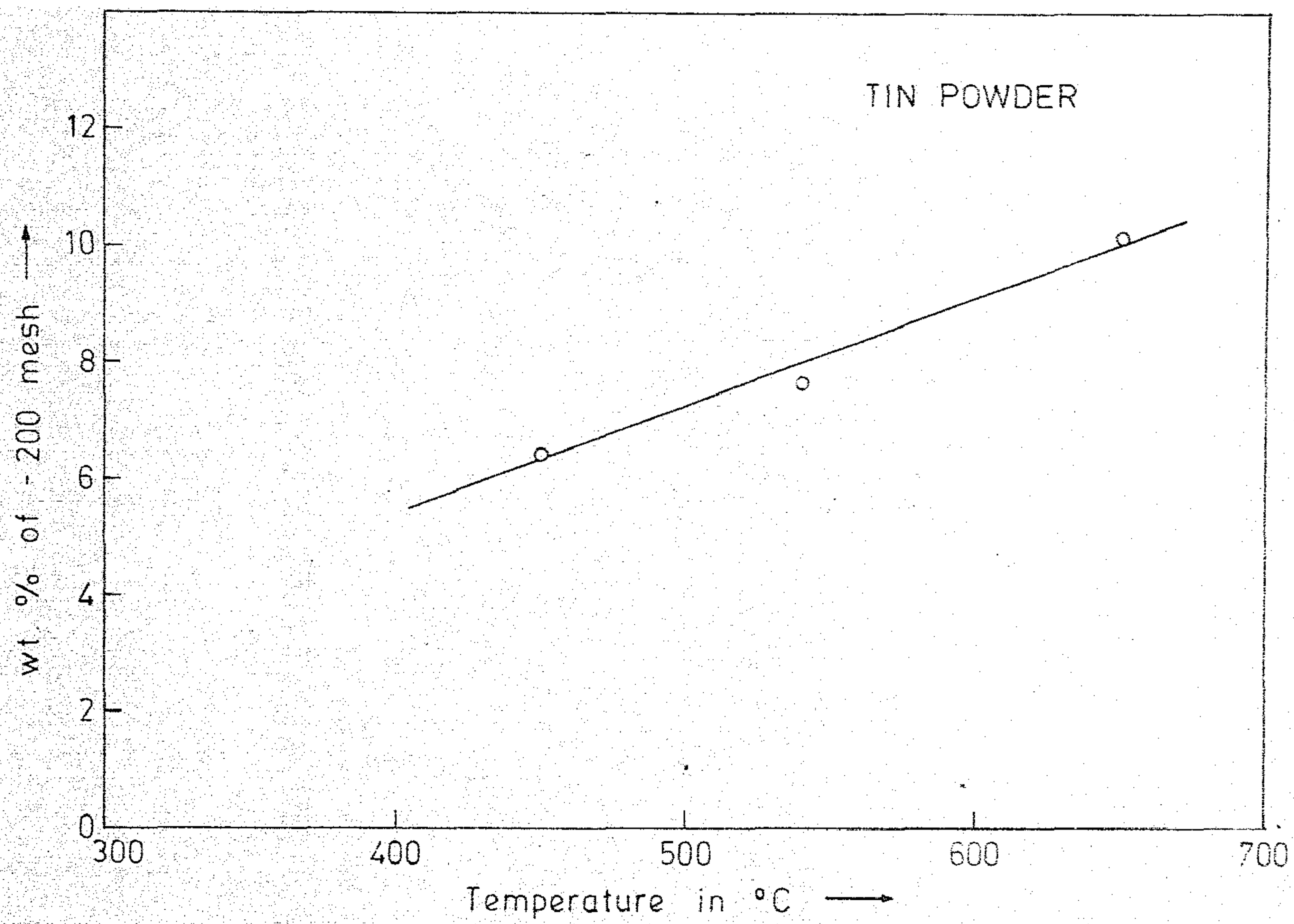

FIG.4.5. EFFECT OF TEMPERATURE ON WEIGHT PERCENTAGE OF -200 MESH POWDER.

Table 4.4

Effect of temperature on particle size distribution of tin powder

Metal : Tin Medium : Air

Temperature : 540°C Nozzle diameter : 1/16"

Pressure : 100 psi

ASTM Sieve No.	Opening size in microns	Average opening size in mm	Weight % retained	Cummulative wt. % passing
+ 40	+420	0.42	10.430	89.570
- 40+45	-420+350	0.385	13.625	75.945
- 45+60	-350+250	0.300	30.270	45.675
- 60+100	-250+150	0.199	14.960	30.715
-100+120	-150+125	0.174	8.630	22.085
-120+140	-125+105	0.114	6.275	15.810
-140+170	-105+88	0.096	4.870	10.940
-170+200	- 88+77	0.081	3.370	7.570
-200+230	- 77+62	0.068	4.730	2.840
-230	- 62	0.062	2.840	-

Average size of powder : 178 microns

Shape : Irregular

Table 4.5

Effect of temperature on particle size distribution of tin powder

Metal : Tin

Medium : Air

Temperature : 450°C

Nozzle diameter : 1/8"

Pressure : 100 psi

ASTM Sieve No.	Opening size in microns	Average opening size in mm	Weight % retained	Cummulative wt. % passing
+ 40	+420	0.42	11.265	89.215
- 40+45	-420+350	0.385	14.370	73.585
- 45+60	-350+250	0.300	32.740	40.845
- 60+100	-250+150	0.199	18.380	23.525
-100+120	-150+125	0.174	5.270	18.255
-120+140	-125+105	0.114	4.150	14.105
-140+170	-105+88	0.096	3.690	10.415
-170+200	- 88+77	0.081	4.175	6.240
-200+230	- 77+62	0.068	3.350	2.890
-230	- 62	0.062	2.600	-

Average size of powder : 196 microns

Shape : Irregular

decreases. Figure 4.4 represents a plot of cummulative weight percent passing versus ASTM size. This plot also supports the view that the amount of fine fraction increases with an increase in temperature of the molten metal. Figure 4.5 shows a plot of weight percent of -200 mesh powder versus temperature of molten metal.

Table 4.1

Temperature in °C	Average size in microns	Wt. % of - 200 mesh
450	196	6.240
540	178	7.570
650	166	10.085

From the Figure 4.5 and Table 4.1 it is obvious that the average particle size decreases and the weight percentage of -200 mesh powder increases with increasing temperature. Lead powder produced under same operating condition was found to be much coarser compared to tin powder. In the case of lead -200 mesh fraction was completely missing, as is evident from Tables 4.7 and 4.8. There are many reasons possible for the particles to be coarse in the case of lead. The density of molten

lead is much higher than that of tin. Therefore, the atomizing pressure required to produce powder particles of same size would be much higher than that in the case of tin. Higher viscosity, and oxidation could be the reasons for production of coarser particles in the case of lead.

4.1.3 Effect of Nozzle Diameter:

Effect of nozzle diameter on size distribution is tabulated in Tables 4.7 and 4.8 respectively. The size distribution plots for the powders produced using two nozzles are shown in Figures 4.6 and 4.7. From the figures it is evident that the size distribution tends to shift towards finer size with decrease in nozzle diameter. The average particle sizes of tin powder were 205 and 196 microns, respectively, for the nozzles of diameter 5/64" and 1/16". In the case of lead the average size for the two nozzles was found to be 198 and 219 microns respectively.

4.2 <u>Flow Rate Test</u>:

The test was carried out as described in Chapter 3 and the results are tabulated in Table 4.9. It can be seen from the table that nitrogen atomized powder exhibited higher flow rate as compared to air atomized powder. This is attributed to the fact that nitrogen

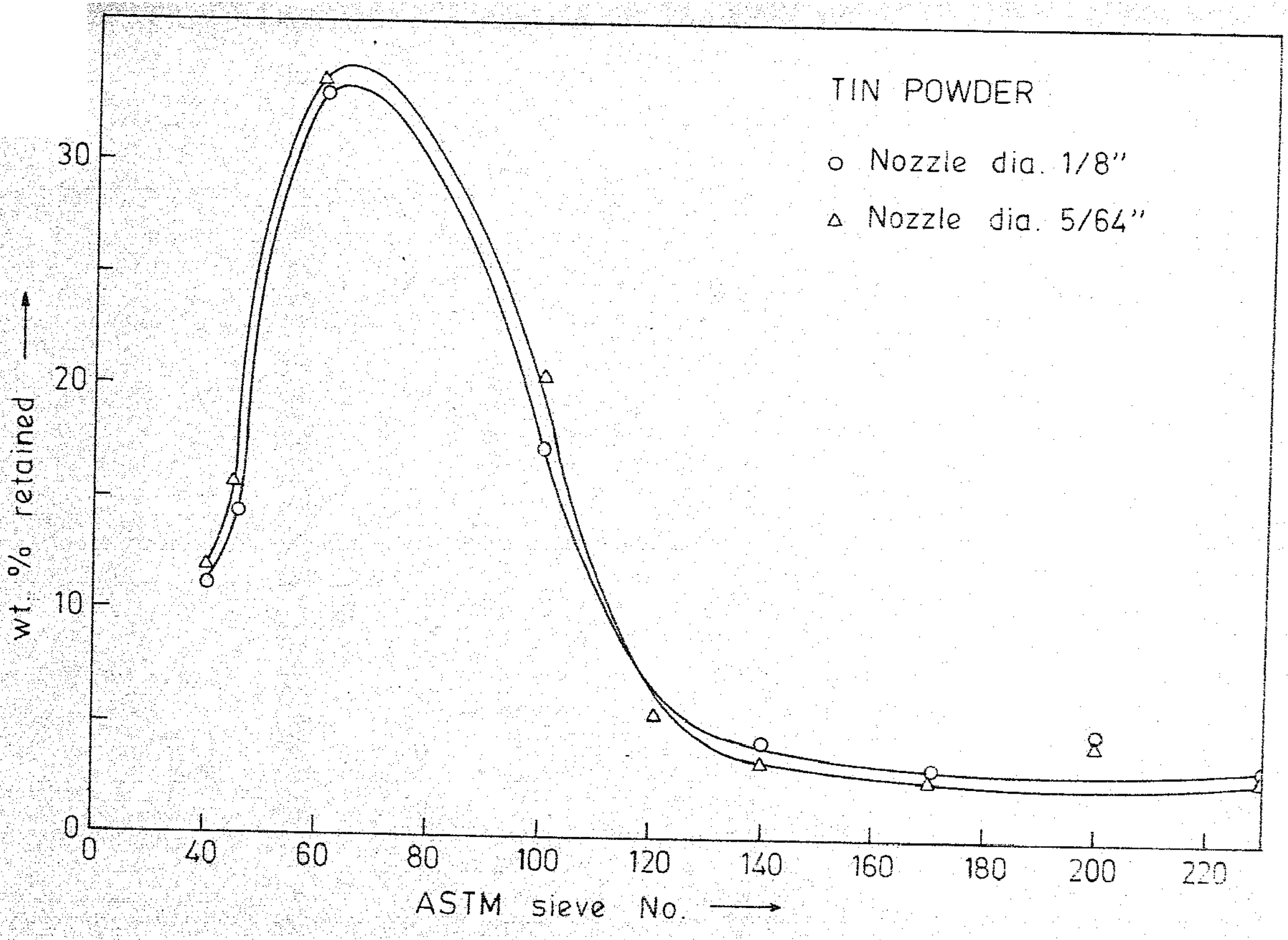

FIG 4.6. EFFECT OF NOZZLE DIAMETER ON PARTICLE SIZE DISTRIBUTION

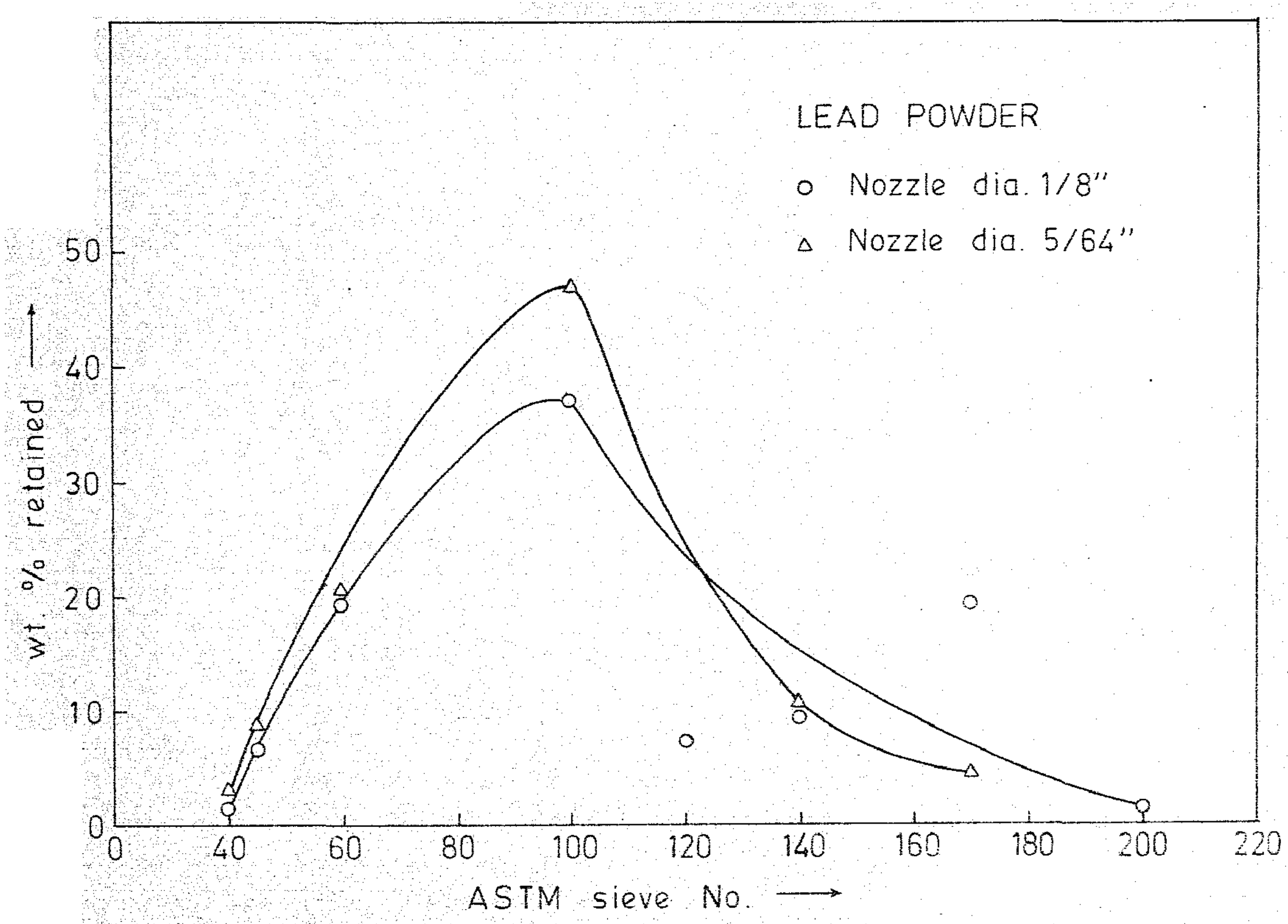

FIG. 4.7. EFFECT OF NOZZLE DIAMETER ON PARTICLE SIZE DISTRIBUTION

Table 4.6

Effect of nozzle diameter on particle size distribution of lead powder

Metal	: Tin	Medium	: Air
Temperature	: 450°C	Nozzle diameter	: 5/64"
Pressure	: 100 psi		

ASTM Sieve No.	Opening size in microns	Average opening size in mm	Weight % retained
+ 40	+420	0.42	11.780
− 40+45	−420+350	0.385	15.630
− 45+60	−350+250	0.300	33.550
− 60+100	−250+150	0.199	19.550
−100+120	−150+125	0.174	5.450
−120+140	−125+105	0.114	3.350
−140+170	−105+88	0.096	2.770
−170+200	− 88+77	0.081	3.240
−200+230	− 77+62	0.068	2.950
−230	− 62	0.062	1.500

Average size of powder : 205 micron

Shape : Irregular

Table 4.7

Effect of nozzle diameter on particle size distribution of lead powder

Metal : Lead   Medium : Air

Temperature : 450°C   Nozzle diameter : 1/6"

Pressure : 100 psi

ASTM Sieve No.	Opening size in microns	Average opening size in mm	Weight % retained
+ 40	+420	0.42	1.230
− 40+45	−420+350	0.385	5.159
− 45+60	−350+250	0.300	19.340
− 60+100	−250+150	0.199	37.420
−100+120	−150+125	0.174	7.290
−120+140	−125+105	0.114	9.050
−140+170	−105+88	0.096	19.160
−170+200	− 88+77	0.081	1.354
−200+230	− 77+62	0.068	−
−230	− 62	0.062	−

Average size of powder : 198 micron

Shape : Irregular

Table 4.8

Effect of nozzle diameter on particle size distribution of lead powder

Metal : Lead Medium : Air

Temperature : 450°C Nozzle diameter : 5/64"

Pressure : 100 psi

ASTM Sieve No.	Opening size in microns	Average opening size in mm	Weight % retained
+ 40	+420	0.420	2.6
- 40+45	-420+350	0.385	8.33
- 45+60	-350+250	0.300	20.00
- 60+100	-250+150	0.199	47.60
-100+120	-150+125	0.174	7.58
-120+140	-125+105	0.114	9.04
-140+170	-105+88	0.096	4.85
-170+200	- 88+74	0.081	-
-200+230	- 74+62	0.068	-
-230	- 62	0.062	-

Average size of powder : 219 micron

Shape : Irregular

atomized particles are more regular in shape compared to irregular particles obtained using air as the atomizing medium. Irregularly shaped particles had tendency to form a bridge, which hindered the flow of metal particles. It was not possible to carry out test for lead powder, for the particles were coarser and very much more irregular in shape. During the test these particles formed bridge and completely blocked the small opening of the flow meter.

4.3 Apparent Density Test:

The results of this test are tabulated in Table 4.9. From the test data it is obvious that the apparent density of tin powder produced using nitrogen as an atomizing medium was highest. This can be explained by the fact these powder particles had a definite regular shape and therefore had a better flowability to fill up the voids within the standard cup whereas the irregular particles tended to form bridge leaving large void space. For air atomized particles apparent density showed a small increase with increase in temperature, perhaps, because of increased amount of finer particles which could fill the voids.

Table 4.9

Flow rate, apparent density and tap density of tin powder

Sl. No.	Atomization temperature	Medium for atomization	Flow rate in seconds	Apparent density gm/cc	Tap density gm/cc
1.	650°C	Nitrogen	22.1	3.325	3.934
2.	650°C	Air	27.5	2.995	3.547
3.	540°C	Air	49.3	2.865	3.298
4.	450°C	Air	54.4	2.761	3.232

4.4 Tap Density:

Results of this test are tabulated in Table 4.9. The tap density was always higher than the corresponding apparent density which was quite logical. The effect of atomizing medium and temperature of molten metal on tap density was the same as the effect of these variables on the apparent density, namely, the tap density of nitrogen atomized powder was greater than tap density of air atomized powder. Not much change in tap density was observed with increase in temperature of molten metal.

4.5 Microscopic Examination:

Powders produced under different operating conditions, were microscopically examined to determine the shape of the particles.

4.5.1 Effect of Atomizing Medium on Shape of the Particle:

Figures 4.8 and 4.9 show air and nitrogen atomized tin particles, respectively, powders of size 420 microns at a magnification of 25X. Figures 4.10 and 4.11 show a similar photographs for powders of 149 microns. Similar trend in change in shape of particles varying fineness with change in atomizing media was observed. On the basis of this observation it can be generalized that,

Fig. 4.8 Tin powder, air atomized at 650°C and 100 psi pressure. Size : 420 microns at magnification of 25X

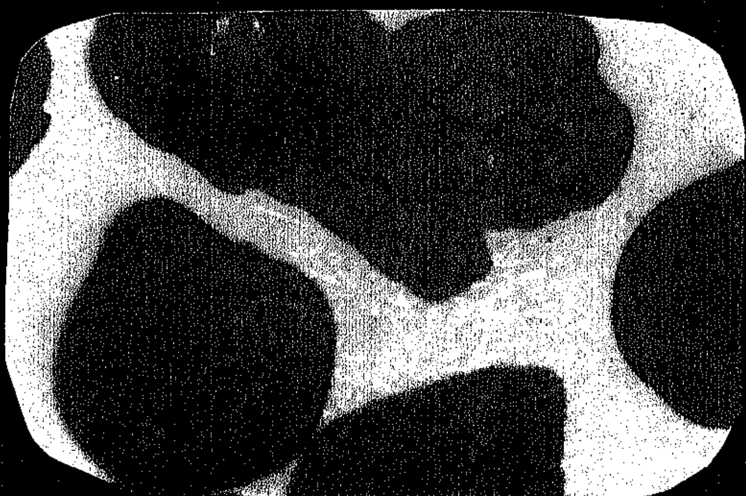

Fig. 9 Tin powder, nitrogen atomized at 650°C and 100 psi pressure. Size: 420 microns at magnification of

Fig. 4.10 Tin powder, air atomized at 650°C and 100 psi pressure. Size: 149 microns at magnification of

Fig. 4.11 Tin powder, nitrogen atomized at 650°C and 100 psi pressure. Size: 149 microns at magnification of

nitrogen atomized powders were more regular in shape compared to air atomized powder of the same fineness. Irregularity in the shape of air atomized powders was perhaps due to rapid oxidation of metal droplets which resulted in formation of thin oxide film on the surface of each droplet. This oxide film once formed did not allow the surface tensional forces to act to make the particles spherical in shape. On the other hand, nitrogen as an atomizing medium did not form any oxide coating and therefore surface tensional forces had enough time to act on a particle to make it more regular in shape. However, the effect of the atomizing medium becomes less and less dominant with increasing fineness of the particles.

4.5.2 Effect of Temperature:

No significant change in shape of powder with increase in metal temperature was observed. In the case of tin powder shape of particles produced from molten metal at three different temperatures namely 650°C, 540°C, and 450°C, was more or less same. This observation is in agreement with the observation cited in literature 8, 13.

CHAPTER 5

CONCLUSION

On the basis of results and discussion of the previous chapter following conclusions can be drawn:

(i) With increase in temperature of molten metal, the size distribution plot moved towards finer size showing an increase in the weight percentage of fine particles. The average size of the particles also decreased with increase in temperature.

(ii) Nitrogen, as an atomizing medium, tended to give finer and more regular particles as compared to air atomized particles.

(iii) Nozzle diameter also affected the size of the particles, a nozzle with smaller diameter shifted the size distribution curve towards the finer size.

(iv) In general, air atomized lead particles were coarser and more irregular in shape compared to tin powders particles produced under similar operating conditions.

(v) Flowability, apparent density and tap density of nitrogen atomized particles were higher than the corresponding values of air atomized particles.

It may be noted that the conclusions that have been drawn here are based on data which is by no means large enough. These conclusions are to be further substantiated by more experimentation.

CHAPTER 6

SUGGESTIONS FOR FUTURE WORK

The existing set-up needs some modifications which are discussed below:

(i) The compressor did not give a constant pressure of air throughout the trial. Therefore, it is necessary to have a compressor which supplies air at constant pressure throughout the run.

(ii) The atomiser design needs a modification. During atomization, some of the atomized particles have a tendency to move upwards immediately after the disintegration of stream of molten metal at impingement point. This is a result of splashing of molten metal due to the high pressurized air jets (see Figure 6.1). These droplets tend to stick to the cold surface of atomizer, and get solidified. This solidification progresses to such an extent that it blocks the opening of the atomizer at the bottom and thereby stops the flow of metal, and hence the atomization process as a whole. It should be possible to take care of this problem by increasing the diameter of atomiser from 'd' to 'D' as shown in Figure 6.2.

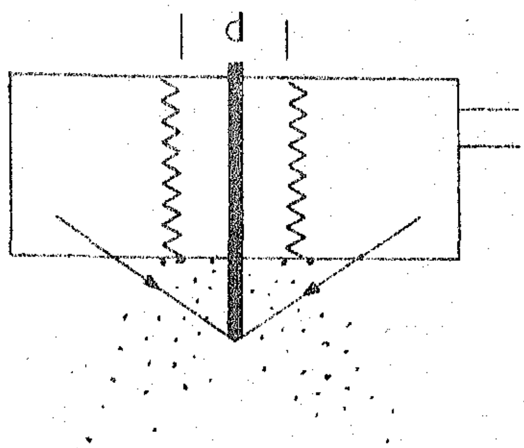

FIG.6.1. Existing atomizer design.

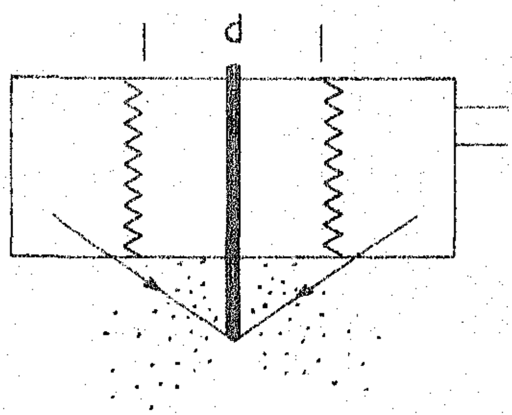

FIG.6.2. Suggested atomizer design.

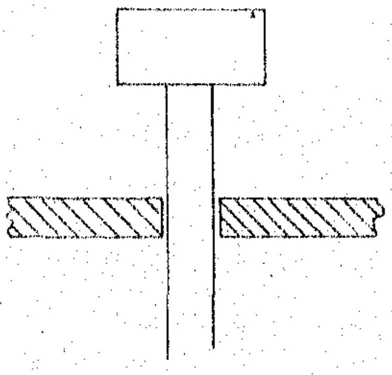

FIG.6.3. Existing design.

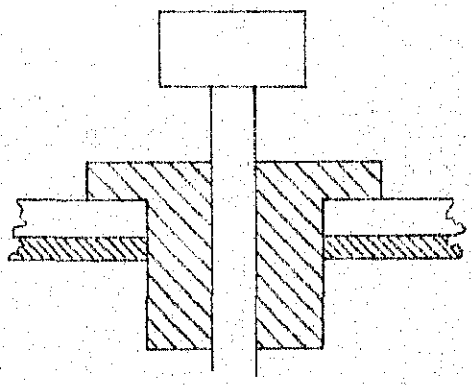

FIG.6.4. Proposed design.

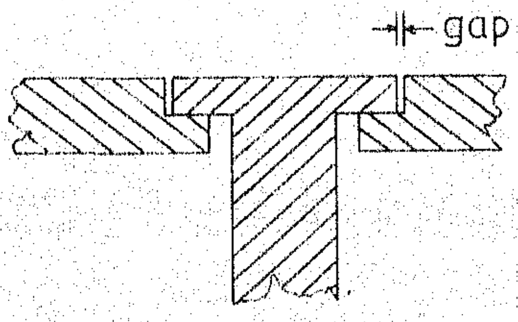

FIG.6.5. Nozzle seat.

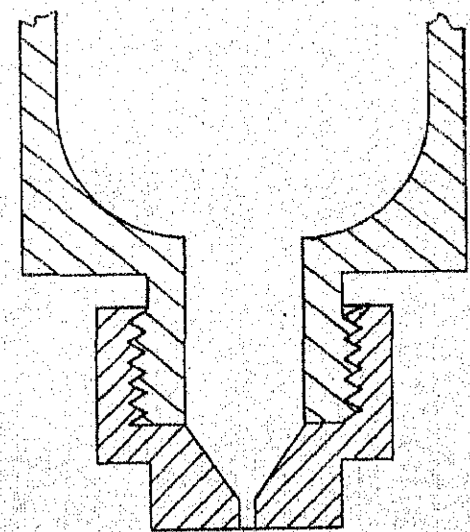

FIG.6.6. Suggested assembly of crucible and nozzle.

(iii) The stopper rod does not have any guide, once it is lifted it does not sit into its position in the nozzle. This create a difficulty in controlling the flow of molten metal.

(iv) The existing and proposed designs are shown in Figures 6.3 and 6.4, respectively.

(v) As we have seen in Figure 3.9 the nozzle sits in the crucible bottom, at the end of the run a small amount of metal is always left in the crucible which is unable to flow down through the nozzle due to high surface tension and viscosity of molten metal. As a result of this small amount of metal always gets solidified at the bottom after the end of the run. Next run cannot be had unless this metal is removed, for the set-up is to be properly aligned before each run. This requires removal and cleaning of the nozzle after every run. Thus if we remove the nozzle after every trial the gap between nozzle and crucible widens (see Figure 6.5), as a result of which molten metal can easily enter the gap. This is dangerous. For this reason it is desirable to have a stainless steel crucible with removable bottom which in itself acts as a nozzle (see Figure 6.6)

The experimental data in the present study is by no means sufficient to draw specific conclusions with confidence. Therefore it is necessary to carry out many more experiments at various operating condition for conformative results.

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